

CAAP Quarterly Report

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Prepared for: *U.S. DOT Pipeline and Hazardous Materials Safety Administration*

Project Title: Development of New Multifunctional Composite Coatings for Preventing and Mitigating Internal Pipeline Corrosion

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For quarterly period ending: *Jun. 30th, 2018*

Business and Activity Section

(a) Generated Commitments

No changes to the existing agreement.

Some purchase made for nano-materials and Q-panels over this reporting period.

(b) Status Update of Past Quarter Activities

The research activities in the 7th quarter continuing efforts by characterizing the hybrid nano-modified coatings and assess their long-term performance, as summarized below.

Tasks 4 and 5: Summary of Characterization of the new coating systems and performance assessment

5.1 Objectives in the 7th Quarter

The following objectives are included in this report: first, bulk density of nanofiller in the solvent; second, adhesion improvement by changing the application method; third, further investigation of the nanocomposite with varied nanofiller content; and water repellency improvement that contributed by varied grades of surface modification.

Meanwhile, the high school students from North Dakota Governor's High School program are invited to participate in our research work during this summer. Both lecture and lab work have been incorporating with this outreach, and this unique opportunity allows us to encourage the next-generation engineers to pursue this research area in the future.

5.2 Experimental Program in the 7th Quarter

5.2.1 Experimental design

The strategy of the experimental study in this report is presented in this section. The study includes the following objectives: characterization of chemical structure by using FTIR analysis, nanofiller volume concentration, performance of the composite with various concentrations. Unless changes were specified in the following section, the sample preparation procedures and test methods from the previous study were continually used in this study.

5.2.1 The bulk density of nanofiller in organic solvent

In the previous study, weight concentration was used to design the nanofiller reinforced composite. However, it has been mentioned that the pigment volume concentration should also be considered as a variable in the experiment study as the density of the nanofillers are various. In order to calculate the filler volume concentration, the density of each nanoparticle in the organic solvent should be measured as the bulk density can be used to represent the relation between volume and weight of nanoparticles in the solvent, as illustrated in Figure 1.



Figure 1. 50 ml measuring flask for the density test

5.2.2 Fabrication of air sprayed specimens

It has been noticed that the major drawback for the tested nanocomposite is the reduction of adhesion strength between coating and substrate. Without changing the content of the dried coating, changing the coating application method from to air spray could be an effective approach as it will provide a more uniformly coating with less air and defects. The nanocomposite was selected to spray with a high volume low pressure spray gun. Adhesion, EIS, and contact angle tests were performed to characterize the sprayed samples.

5.2.3 Fabrication of the specimens with varying filler concentrations

In the section, the concentration of nanofiller was controlled. Considering the wide range of concentration in tested samples, two masterbatches were prepared. Corrosion barrier performance, abrasion resistance, contact angle, adhesion and dogbone tensile test were performed to characterize the prepared samples.



Figure 2. Typical nanocomposites

5.3 Results and Discussions

5.3.1 The bulk density of fillers in organic solvent

The measurement results of bulk density for each type of nanofiller were summarized. The significant variation between the true density and bulk density. Generally, the value of bulk density is smaller than true density as the inter-particle void volume will be included in the measurement of bulk density. For the hybrid filler, the theoretical density was calculated by taking the average of every single filler. It has been noticed that different values were obtained after measurement, this indicates that the interaction between different types of nanofiller leads to change the inter-particle voids in the nanoparticle network.

5.3.2 The nanocomposites specimens prepared by air sprayed

This section presents the evaluation of adhesion, corrosion barrier effect and the contact angle of the samples that prepared by air spray. To purpose of this study is to improve the adhesive strength between nanocomposites and substrate.

a) Adhesive bonding strength

The pull-off strength (adhesion) was measured by following ASTM D4541 to find out whether the adhesion between the nanocomposites will be improved by changing the application method. The

strength has been significantly improved in certain groups which the coating was air sprayed to the substrate, and the strength (3 MPa) is very close to the neat epoxy group.

b) Corrosion barrier performance: EIS

To evaluate the corrosion protection properties, a comparative study was performed. The impedance curves are presented in Figure 3, and excellent barrier property was still obtained in the nanocomposite 2 group.

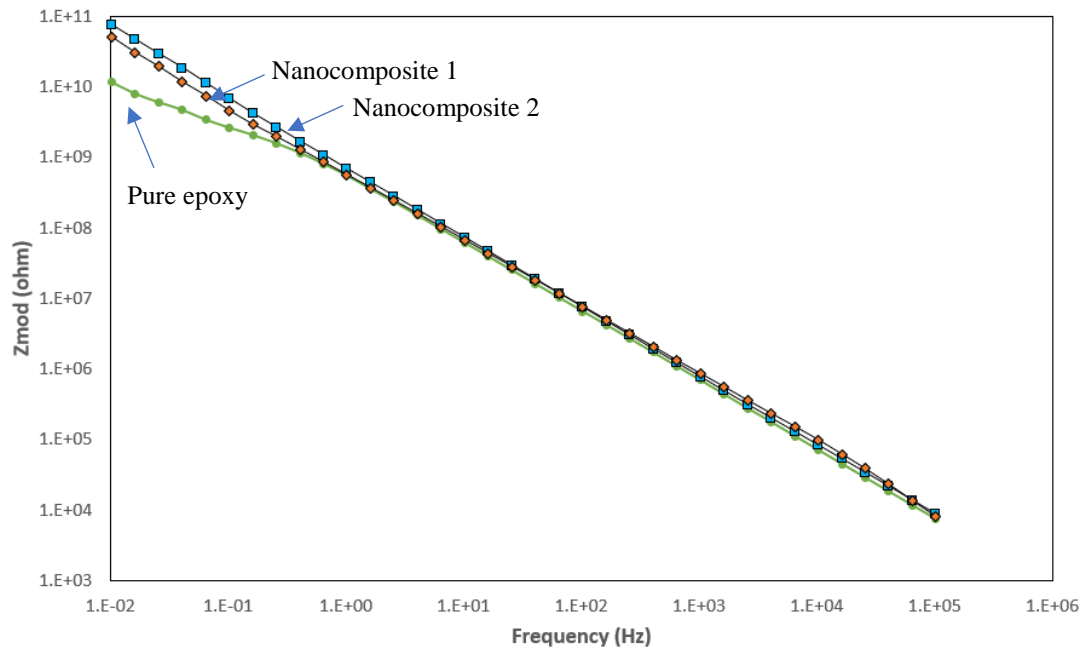


Figure 3. Impedence curve of the air sprayed samples

c) Contact angle test

Figure 4 is showing the contact angle of the samples; it is easy to be observed that there are no significant changes in contact angle for the samples that applied by air sprayed. The water contact angle of the sample is around 25 degrees which are very close to the sample that prepared by drawdown method.

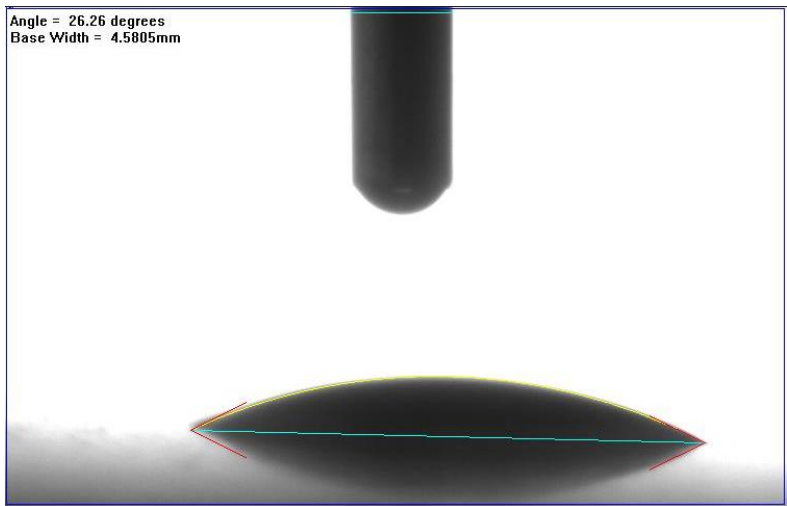


Figure 4. Water contact angle of the air sprayed GSE1 sample

5.3.3 The nanocomposites with varying filler concentrations

The test results of GSE groups with varied concentrations were summarized in this section.

a) Corrosion barrier performance: EIS

To evaluate the effect of the filler concentration on corrosion protection properties in the nanocomposites, the bode curves that obtained from Potentiostatic EIS were summarized in this section. Excellent corrosion barrier effect was observed.

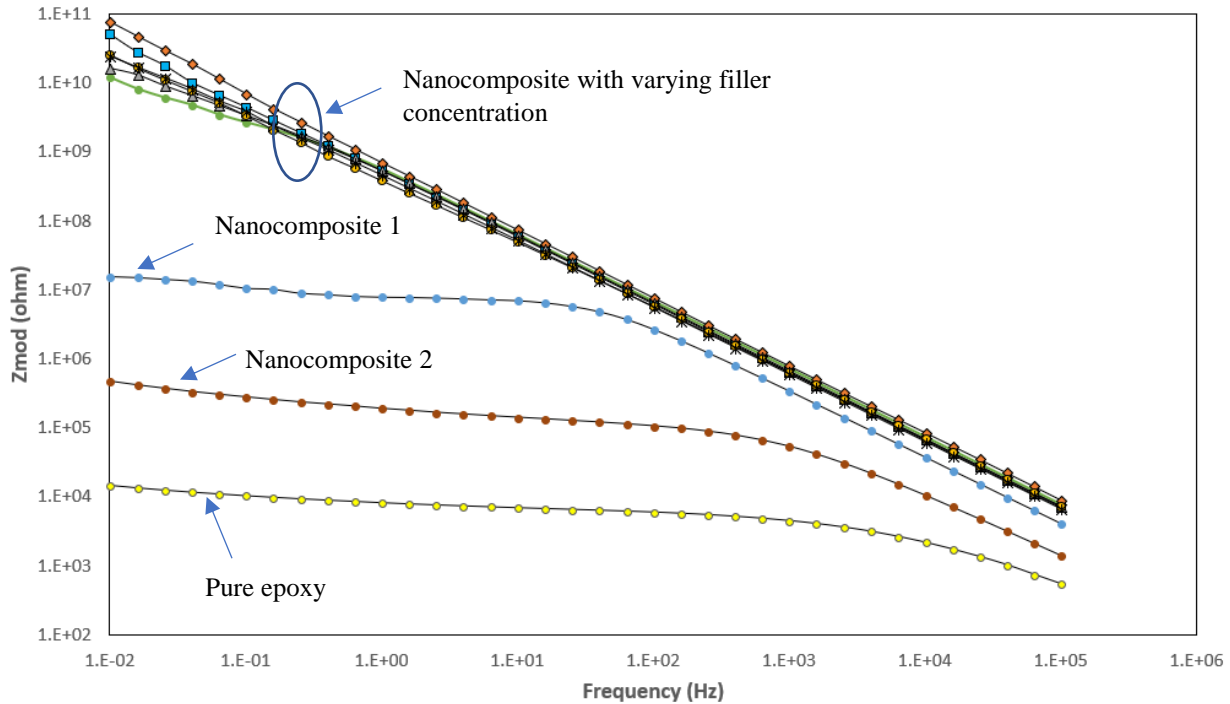


Figure 5. Impedance curve of the nanocomposites with varied filler concentration

b) Abrasion resistance test

The results of abrasion test for the nanocomposites were conducted and improvement of abrasion resistance was obtained in the sample groups.

c) Dogbone tensile test

The tensile property of the groups with varying concentrations was summarized. With the higher content of the nanofiller was incorporated into a composite, the max stress starts to decrease. On the other hand, the results of failure strain are presented. Unlike the tensile stress curve, the failure strain maintained higher than neat epoxy at all the tested samples, and the highest strain was obtained about 39.8% increase.

d) Adhesive bonding strength

The stress was reduced by the incorporation of nanofiller regardless the weight concentration changes. The adhesion between the nanocomposites and substrate maintained in the range of 170 to 250 Psi.

e) Contact angle test

Similar to the nanocomposites that tested in the previous study, regardless of the concentration of the nanofiller, the incorporation of the nanofiller into epoxy resin also results in a reduction of water contact angle.

f) Hydrophobicity of abraded surfaces

Surface modification was applied on nanocomposite with varied concentrations with different abrasive grades. Contact angle test was performed on the abraded surface of the coating, and the test results are summarized. Similar results were observed in all tested fillers with varied concentration and abrasive grades. The contact angle (CA) of all the tested nanocomposites was remarkably increased after the surface modifications.

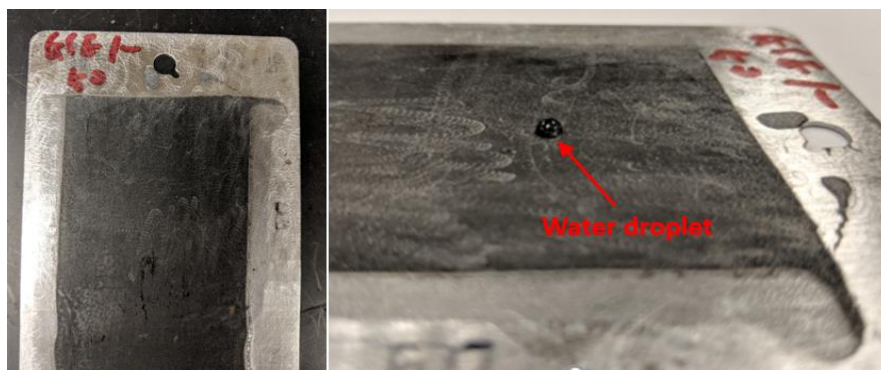


Figure 6. Water contact angle of the samples after surface modification

5.3.5 Mentoring students from North Dakota Governor's Schools Programs

In this summer, as illustrated in Figure 7, four high school students from North Dakota Governor's Schools program were invited to experience the research in our research group.



Figure 7. High schools working with Dr. Lin's research group for preparing coating samples and performing tests.

5.3.6 Summary

In summary, several findings can be revealed by the results of our experimental program:

- Significant variation between true density and bulk density can be observed for nanofillers as the value of the inter-particle void volume will be included in the measurement of bulk density. Bulk density will be used to determine the volume concentration for the nanocomposites.
- For the nanocomposites, the air sprayed sample has stronger adhesion between coating and substrates compare with the samples that prepared by drawdown method.
- Excellent corrosion barrier effect was observed. However, dramatic reduction of impedance value was observed once the concentration of the nanofiller increase too high.
- Water contact angle was significantly increased by all the tested samples with different abrasive grades.

5.3.7 Future work

The following targets will be focused on the next step based on the experimental study in this report:

- To evaluating the long-term performance of the hybrid nanocomposites, the salt spray test (ASTM B117) should be performed to determine the corrosion protection properties changes due to the addition of nanofillers.
- For the hybrid rcomposites, further investigation should be conducted for evaluating the performance.
- The incorporation of nanofillers should be considered if positive results were obtained in the hybrid polymer from the previous step.